Tetraquinolinium ditelluro(VI)octavanadate(V) octahydrate

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In the title compound, $(C_9H_8N)_4[Te_2V_8O_{28}]\cdot8H_2O$, the complete heteropolyanion is generated by a crystallographic inversion centre. One of the two quniolinium ions forms an $N-H\cdots O_p$ (p = polyoxidometallate) hydrogen bond and the other an $N-H\cdots O_w$ (w = water) hydrogen bond. The water molecules further link the components by $O-H\cdots O_p$ and $O-H\cdots O_w$ hydrogen bonds. A number of $C-H\cdots O$ interactions and aromatic $\pi-\pi$ stacking interactions [shortest centroid–centroid separation = 3.541 (7) Å] are also observed. Together, these generate a three-dimensional network.

Related literature

For applications of polyoxidometallates, see: Fukuda & Yamase (1997); Rajakumar *et al.* (2000); Folbergrova & Mares (1987); Fantus *et al.* (1995). For bond-valence calculations, see: Brown & Altermatt (1985). For geometrical features in related structures, see: Lee *et al.* (2008); Joo *et al.* (2011); Strukan *et al.* (1997); Konaka *et al.* (2008, 2011); Evans *et al.* (1966); Hemissi *et al.* (2010).
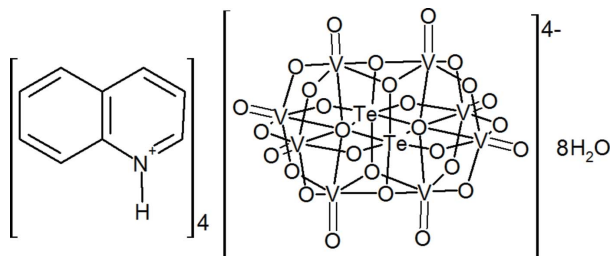
Experimental

Crystal data

$(C_9H_8N)_4[Te_2V_8O_{28}]\cdot8H_2O$	Triclinic, $P\bar{1}$
$M_r = 1775.50$	$a = 10.907$ (3) Å
$b = 11.302$ (3) Å	$Z = 1$
$c = 13.169$ (2) Å	Ag $K\alpha$ radiation
$\alpha = 106.45$ (4)°	$\lambda = 0.56087$ Å
$\beta = 107.71$ (4)°	$\mu = 1.28$ mm^{-1}
$\gamma = 105.34$ (4)°	$T = 295$ K
$V = 1369.4$ (6) Å^3	$0.19 \times 0.15 \times 0.09$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	13245 independent reflections
Absorption correction: analytical (Alcock, 1970)	6549 reflections with $I > 2\sigma(I)$
$T_{min} = 0.561$, $T_{max} = 0.725$	$R_{int} = 0.045$
16455 measured reflections	2 standard reflections every 120 min intensity decay: 4%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.115$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.308$	
$S = 1.05$	
13245 reflections	$\Delta\rho_{max} = 3.11$ e Å^{-3}
385 parameters	$\Delta\rho_{min} = -2.28$ e Å^{-3}
20 restraints	

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H2W1···O2^i	0.85 (1)	1.82 (1)	2.663 (8)	178 (1)
O1W—H1W1···O7	0.85 (1)	1.92 (1)	2.762 (9)	170 (4)
O2W—H1W2···O8^{ii}	0.85 (1)	2.03 (2)	2.842 (10)	159 (4)
O2W—H2W2···O4W^{iii}	0.85 (1)	2.10 (1)	2.952 (14)	177 (1)
O3W—H2W3···O4W	0.84 (1)	1.91 (1)	2.754 (14)	177 (1)
O3W—H1W3···O6^{ii}	0.85 (1)	1.83 (2)	2.665 (11)	166 (6)
O4W—H2W4···O1W^{iv}	0.85 (1)	2.41 (1)	2.826 (12)	111 (3)
O4W—H1W4···O2W	0.85 (1)	2.26 (5)	2.836 (17)	125 (5)
N1—H1···O1W	0.86	1.85	2.700 (11)	172
N2—H2···O14	0.86	1.88	2.740 (9)	175
C5—H5···O6^{ii}	0.93	2.29	3.180 (13)	160
C6—H6···O13^v	0.93	2.48	3.178 (14)	132
C7—H7···O1^{vi}	0.93	2.56	3.350 (13)	143
C7—H7···O2W^{vii}	0.93	2.57	3.296 (14)	135
C10—H10···O9^{viii}	0.93	2.51	3.275 (13)	140
C14—H14···O4^{ix}	0.93	2.58	3.403 (14)	148
C17—H17···O5	0.93	2.60	3.411 (13)	146

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $x+1, y, z+1$; (iii) $-x+2, -y, -z+2$; (iv) $x+1, y, z$; (v) $-x+1, -y+1, -z+2$; (vi) $x, y-1, z$; (vii) $x-1, y, z$; (viii) $-x+1, -y+1, -z+1$; (ix) $-x+1, -y+2, -z+1$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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supporting information

Tetraquinolinium ditelluro(VI)octavanadate(V) octahydrate

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S1. Comment

The polyoxovanadate materials (POVs) are well established in magnetic, electric and biomedical fields because of their richness structural variety in relation with the vanadium element which can adopt different coordination geometries and variable oxidation states very interesting for redox applications in catalysis and materials science (Fukuda *et al.*, 1997; Rajakumar *et al.*, 2000; Folbergrova *et al.*, 1987; Fantus *et al.*,1995). Furthermore, this family is dominated by the decavanadates, well known in their protonated forms $[H_xV_{10}O_{28}]^{(6-x)-}$. Nevertheless, incorporation of other elements besides vanadium into this structural type has not been reported yet. In particular, substitution of one or more vanadium atoms by Pt^{IV} (Lee *et al.*, 2008; Joo *et al.*, 2011), Mo^{VI}(Strukan *et al.*, 1997) and Te^{VI} (Konaka *et al.*, 2008; Konaka *et al.*, 2011) are investigated and resulted into novel materials with highly promising catalytic properties. To date in our knowledge, only two POVs incorporated one telluric, are known (Konaka *et al.*, 2008). Here, we describe the synthesis and structure of the first ocatavanado-ditellurate ion, $[Te_2V_8O_{28}]^{4-}$, which was isolated as the hydrated quinolinium salt $(C_9H_8N)_4[Te_2V_8O_{28}]\cdot8H_2O$ (I). The asymmetric unit of (I) contains one half of a $[Te_2V_8O_{28}]^{4-}$ anion, two crystallographically independent quinolinium cations and four water molecules. Owing to the inversion symmetry, the whole polyanion is generated, resulting so to the title compound formulae (Fig. 1). The structure of the $[Te_2V_8O_{28}]^{4-}$ polyanion is basically the same as that of decavanadate, $[V_{10}O_{28}]^{6-}$ (Evans *et al.*, 1966). In $[Te_2V_8O_{28}]^{4-}$ anion two of the central V atoms of $[V_{10}O_{28}]^{6-}$ are replaced with two Te atoms. The valence bond calculation (Brown & Altermatt, 1985) gives effective bond valences of 6.1647 for Te cation and of 5.0743, 5.0068, 5.0574 and 5.063 for the four independent V cations, consistent with their oxidation states Te(+VI) and V(+V). Replacement of central V atom with a heteroatom, has also been observed for $[H_2PtV_9O_{28}]^{5-}$ (Lee *et al.*,2008; Joo *et al.*, 2011). As for the decavanadate anion, the $[Te_2V_8O_{28}]^{4-}$ anion is built up of 10 e dge sharing MO_6 (M = V or Te) octahedra. Within VO_6 octahedra, the V—O distances are also similar to those already observed and depend upon the type of oxo ligand: bond lengths to the terminal oxo oxygen V—Ot are between 1.602 (7) and 1.610 (6) Å, V—O2b bond lengths to the oxygen bonded to two V atoms vary from 1.767 (6) e t 1.864 (6) Å, V—O3b bond lengths to the oxygen bonded to three V atoms are 2.005 (7) and 2.039 (7) and finally, V—O6c bond lengths to the oxygen shared between six V atoms are 2.380 (7) and 2.387 (7) Å. The VO_6 octahedra are significantly distorted, with the bond angles at the V atoms ranging from 73.5 (3) to 176.5 (3)°. The TeO_6 octahedra are less distorted in comparison with VO_6 ocathedra since the Te—O distances vary from 1.765 (6) to 2.080 (7) Å. Similar trends are also observable for similar heteropolyanion (Konaka *et al.*, 2008; Konaka *et al.*, 2011). The quinolinium cations exhibit the typical ranges in bond lengths and angles as found in the related structures (Hemissi *et al.*, 2010).

In the crystal packing, the discrete $[Te_2V_8O_{28}]^{4-}$ polyanions are hydrogen bonded through clusters of eight water molecules $[H_2O]_8$ forming layers $[Te_2V_8O_{28}(H_2O)_8]_n^{4n-}$ stacked along the [100] direction (Fig. 2). With regard to the organic moieties, the two crystallographically independent $[C_9H_7—N(1)H]^+$ and $[C_9H_7—N(2)H]^+$ cations are interconnected thanks to intermolecular $\pi\cdots\pi$ stacking interactions with centroid-centroid ring separations between 3.54 (8) and 3.90 (7) Å as to develop chains extending along [011] direction. Furthermore, the quinolinium chains and the

polyanion sheets are linked thanks to O1W, O2W water molecules and terminal oxygen atoms (O1, O5 and O9) and bridged oxygen atoms (μ 2-O6, μ 2-O13, μ 2-O14 and μ 3-O4) of the polyanion, into a three dimensional network by N —H···O and C—H···O hydrogen bonds with donor—acceptor distances ranging from 2.700 (11) to 3.411 (13) Å.

S2. Experimental

Vanadium (V) oxide (1.26 g, 6.93 mmol), quinoline (0.74 ml, 6.16 mmol) and telluric acid $Te(OH)_6$ (0.36 g, 1.55 mmol) were dissolved in a mixture of 30 ml of distilled water and 10 ml of ethanol and then stirred for 3 h. Yellow single crystals were obtained after one week by slow evaporation at room temperature.

S3. Refinement

All H atoms attached to C and N atoms were fixed geometrically and treated as riding, with C—H = 0.93 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2Ueq(C, N) for aromatic rings. The water H atoms were refined using restraints [O—H = 0.85 (1) A °, H···H = 1.44 (2) A ° and U_{iso}(H) = 1.5Ueq(O)].
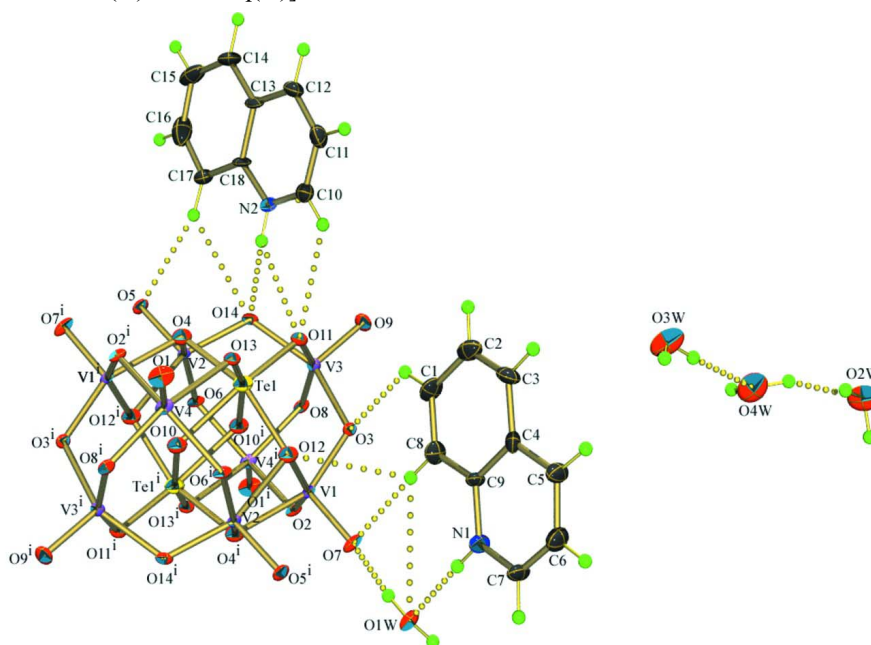
Figure 1

An *ORTEP* view of (I) with displacement ellipsoids drawn at the 30% probability level. Hydrogen bonds are represented as dashed lines. [Symmetry code: (i) 1 - *x*, 1 - *y*, - *z*]
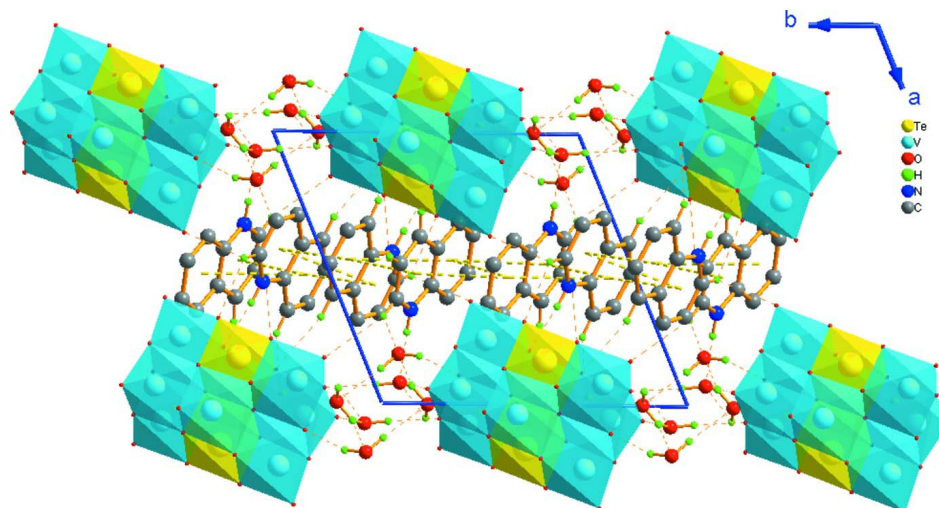
Figure 2

Structure projection of (I) along the [001] direction. The $\pi \cdots \pi$ stacking interaction are represented as yellow dashed lines and the hydrogen bonds by red ones. The H-atoms not involved in H-bonding are omitted.

Tetraquinolinium ditelluro(VI)octavanadate(V) octahydrate

Crystal data

$(C_9H_8N)_4[Te_2V_8O_{28}]\cdot 8H_2O$	$Z = 1$
$M_r = 1775.50$	$F(000) = 868$
Triclinic, $P\bar{1}$	$D_x = 2.153$ Mg m^{-3}
Hall symbol: -P 1	Ag $K\alpha$ radiation, $\lambda = 0.56087$ Å
$a = 10.907\ (3)$ Å	Cell parameters from 25 reflections
$b = 11.302\ (3)$ Å	$\theta = 9–11°$
$c = 13.169\ (2)$ Å	$\mu = 1.28$ mm^{-1}
$\alpha = 106.45\ (4)°$	$T = 295$ K
$\beta = 107.71\ (4)°$	Rectangular, yellow
$\gamma = 105.34\ (4)°$	$0.19 \times 0.15 \times 0.09$ mm
$V = 1369.4\ (6)$ Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	13245 independent reflections
Radiation source: fine-focus sealed tube	6549 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.045$
non–profiled ω scans	$\theta_{max} = 28.0°$, $\theta_{min} = 2.3°$
Absorption correction: analytical (Alcock, 1970)	$h = -18 \rightarrow 17$
	$k = -18 \rightarrow 18$
	$l = -3 \rightarrow 22$
$T_{min} = 0.561$, $T_{max} = 0.725$	2 standard reflections every 120 min
16455 measured reflections	intensity decay: 4%

Refinement

Refinement on F^2	385 parameters
Least-squares matrix: full	20 restraints
$R[F^2 > 2\sigma(F^2)] = 0.115$	Primary atom site location: structure-invariant direct methods
$wR(F^2) = 0.308$	
$S = 1.05$	Secondary atom site location: difference Fourier map
13245 reflections	

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0978P)^2 + 22.5177P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 3.11$ e Å^{-3}
$\Delta\rho_{min} = -2.28$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Te1	0.16225 (7)	0.58886 (7)	0.58253 (6)	0.03140 (18)
V1	0.05217 (14)	0.27420 (12)	0.49481 (12)	0.0177 (2)
V2	0.03656 (13)	0.57033 (13)	0.32457 (11)	0.0166 (2)
V3	0.23652 (14)	0.42319 (14)	0.39532 (13)	0.0213 (3)
V4	0.07774 (16)	0.74944 (13)	0.76322 (12)	0.0215 (3)
O1	0.1649 (8)	0.8702 (7)	0.8882 (6)	0.0356 (16)
O2	−0.0552 (6)	0.1678 (5)	0.3401 (5)	0.0198 (10)
O3	0.2094 (6)	0.3145 (6)	0.4763 (5)	0.0209 (11)
O4	0.1186 (7)	0.6932 (7)	0.4925 (6)	0.03140 (18)
O5	0.0293 (7)	0.6753 (6)	0.2649 (6)	0.0261 (12)
O6	−0.0685 (6)	0.4051 (5)	0.2031 (5)	0.0209 (11)
O7	0.0538 (8)	0.1673 (6)	0.5536 (6)	0.0289 (14)
O8	0.1039 (6)	0.2887 (5)	0.2588 (5)	0.0208 (11)
O9	0.3794 (7)	0.4249 (7)	0.3844 (7)	0.0322 (14)
O10	−0.0326 (7)	0.5676 (7)	0.5778 (6)	0.03140 (18)
O11	0.3065 (6)	0.5744 (6)	0.5518 (5)	0.0233 (11)
O12	0.1318 (7)	0.4462 (7)	0.6351 (6)	0.03140 (18)
O13	0.2328 (6)	0.7205 (6)	0.7204 (5)	0.0215 (11)
O14	0.1984 (6)	0.5518 (6)	0.3384 (5)	0.0203 (10)
O1W	0.0759 (8)	0.0847 (6)	0.7344 (6)	0.0335 (15)
H1W1	0.067 (3)	0.118 (3)	0.683 (2)	0.040*
H2W1	0.0671	0.0039	0.7102	0.040*
O2W	1.1818 (11)	0.1180 (8)	1.1089 (9)	0.060 (3)
H1W2	1.176 (13)	0.185 (2)	1.154 (12)	0.072*
H2W2	1.1280	0.0421	1.0989	0.072*
O3W	0.9613 (14)	0.3830 (8)	1.0059 (9)	0.065 (3)
H1W3	0.947 (18)	0.401 (9)	1.068 (8)	0.078*
H2W3	0.9696	0.3095	0.9801	0.078*
O4W	0.9969 (13)	0.1478 (11)	0.9221 (9)	0.067 (3)
H1W4	1.0634 (19)	0.121 (3)	0.936 (5)	0.081*

H2W4	0.947 (3)	0.1236 (17)	0.8506 (12)	0.081*
N1	0.3469 (8)	0.2174 (8)	0.8833 (7)	0.0351 (18)
H1	0.2632	0.1746	0.8307	0.042*
N2	0.4422 (8)	0.7675 (7)	0.4207 (8)	0.0297 (16)
H2	0.3644	0.7027	0.3982	0.036*
C1	0.4842 (14)	0.4522 (13)	0.7670 (11)	0.046 (3)
H1A	0.4551	0.4753	0.7040	0.056*
C2	0.6262 (15)	0.5203 (13)	0.8545 (13)	0.049 (3)
H2A	0.6894	0.5873	0.8470	0.059*
C3	0.6670 (12)	0.4882 (11)	0.9452 (13)	0.047 (3)
H3	0.7581	0.5342	1.0011	0.056*
C4	0.5747 (10)	0.3857 (10)	0.9581 (9)	0.0305 (18)
C5	0.6157 (12)	0.3427 (12)	1.0502 (11)	0.047 (3)
H5	0.7075	0.3808	1.1049	0.056*
C6	0.5166 (14)	0.2435 (13)	1.0568 (11)	0.047 (3)
H6	0.5410	0.2175	1.1186	0.056*
C7	0.3861 (11)	0.1844 (11)	0.9754 (10)	0.039 (2)
H7	0.3207	0.1189	0.9825	0.046*
C8	0.3937 (12)	0.3527 (12)	0.7797 (10)	0.040 (2)
H8	0.3017	0.3089	0.7251	0.048*
C9	0.4371 (9)	0.3171 (9)	0.8717 (8)	0.0274 (17)
C10	0.5429 (11)	0.7920 (10)	0.5154 (10)	0.036 (2)
H10	0.5287	0.7373	0.5553	0.043*
C11	0.6672 (12)	0.8923 (12)	0.5600 (12)	0.048 (3)
H11	0.7361	0.9090	0.6306	0.057*
C12	0.6909 (11)	0.9708 (11)	0.4981 (12)	0.043 (3)
H12	0.7776	1.0383	0.5247	0.052*
C13	0.5836 (10)	0.9467 (8)	0.3967 (11)	0.038 (2)
C14	0.5994 (13)	1.0189 (12)	0.3266 (13)	0.049 (3)
H14	0.6845	1.0867	0.3490	0.059*
C15	0.4871 (19)	0.9884 (14)	0.2232 (16)	0.063 (4)
H15	0.4970	1.0390	0.1793	0.076*
C16	0.3654 (17)	0.8868 (15)	0.1870 (15)	0.066 (4)
H16	0.2949	0.8661	0.1159	0.080*
C17	0.3395 (11)	0.8109 (10)	0.2506 (11)	0.038 (2)
H17	0.2524	0.7452	0.2267	0.046*
C18	0.4533 (9)	0.8398 (8)	0.3537 (9)	0.0289 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Te1	0.0292 (3)	0.0266 (3)	0.0309 (3)	0.0036 (2)	0.0090 (2)	0.0112 (2)
V1	0.0244 (6)	0.0124 (5)	0.0195 (6)	0.0075 (4)	0.0102 (5)	0.0092 (5)
V2	0.0188 (5)	0.0153 (5)	0.0169 (6)	0.0046 (4)	0.0073 (5)	0.0100 (5)
V3	0.0195 (6)	0.0223 (6)	0.0271 (7)	0.0087 (5)	0.0126 (5)	0.0129 (5)
V4	0.0271 (7)	0.0146 (5)	0.0144 (6)	0.0031 (5)	0.0058 (5)	0.0018 (4)
O1	0.046 (4)	0.027 (3)	0.017 (3)	0.003 (3)	0.009 (3)	0.000 (2)
O2	0.027 (3)	0.012 (2)	0.019 (3)	0.0035 (19)	0.009 (2)	0.0077 (19)

O3	0.020 (2)	0.022 (3)	0.024 (3)	0.008 (2)	0.009 (2)	0.014 (2)
O4	0.0292 (3)	0.0266 (3)	0.0309 (3)	0.0036 (2)	0.0090 (2)	0.0112 (2)
O5	0.030 (3)	0.025 (3)	0.029 (3)	0.011 (2)	0.011 (3)	0.020 (3)
O6	0.023 (3)	0.011 (2)	0.017 (2)	−0.0030 (18)	0.004 (2)	0.0032 (18)
O7	0.050 (4)	0.018 (3)	0.023 (3)	0.014 (3)	0.016 (3)	0.013 (2)
O8	0.029 (3)	0.017 (2)	0.016 (2)	0.007 (2)	0.009 (2)	0.008 (2)
O9	0.027 (3)	0.042 (4)	0.040 (4)	0.017 (3)	0.022 (3)	0.022 (3)
O10	0.0292 (3)	0.0266 (3)	0.0309 (3)	0.0036 (2)	0.0090 (2)	0.0112 (2)
O11	0.018 (2)	0.025 (3)	0.024 (3)	0.003 (2)	0.008 (2)	0.011 (2)
O12	0.0292 (3)	0.0266 (3)	0.0309 (3)	0.0036 (2)	0.0090 (2)	0.0112 (2)
O13	0.017 (2)	0.019 (2)	0.022 (3)	0.0013 (19)	0.006 (2)	0.008 (2)
O14	0.017 (2)	0.023 (3)	0.019 (3)	0.0009 (19)	0.007 (2)	0.011 (2)
O1W	0.047 (4)	0.022 (3)	0.028 (3)	0.013 (3)	0.009 (3)	0.013 (3)
O2W	0.080 (7)	0.051 (5)	0.058 (6)	0.021 (5)	0.051 (6)	0.013 (5)
O3W	0.110 (9)	0.050 (5)	0.043 (5)	0.027 (6)	0.045 (6)	0.019 (4)
O4W	0.095 (8)	0.075 (7)	0.052 (6)	0.037 (6)	0.045 (6)	0.032 (5)
N1	0.029 (4)	0.030 (4)	0.031 (4)	0.001 (3)	0.001 (3)	0.013 (3)
N2	0.028 (3)	0.016 (3)	0.040 (4)	0.004 (3)	0.012 (3)	0.009 (3)
C1	0.063 (8)	0.053 (7)	0.041 (6)	0.026 (6)	0.027 (6)	0.033 (6)
C2	0.062 (8)	0.040 (6)	0.065 (8)	0.022 (6)	0.045 (7)	0.025 (6)
C3	0.028 (5)	0.033 (5)	0.067 (8)	0.000 (4)	0.021 (5)	0.013 (5)
C4	0.030 (4)	0.030 (4)	0.030 (5)	0.017 (4)	0.007 (4)	0.010 (4)
C5	0.030 (5)	0.042 (6)	0.050 (7)	0.000 (4)	−0.001 (5)	0.024 (5)
C6	0.051 (7)	0.046 (6)	0.043 (6)	0.017 (5)	0.012 (5)	0.025 (5)
C7	0.034 (5)	0.039 (5)	0.037 (5)	0.002 (4)	0.009 (4)	0.024 (4)
C8	0.040 (5)	0.046 (6)	0.028 (5)	0.014 (5)	0.007 (4)	0.016 (4)
C9	0.026 (4)	0.027 (4)	0.030 (4)	0.004 (3)	0.013 (3)	0.016 (3)
C10	0.037 (5)	0.030 (4)	0.043 (6)	0.015 (4)	0.020 (5)	0.012 (4)
C11	0.030 (5)	0.043 (6)	0.059 (8)	0.016 (5)	0.008 (5)	0.014 (6)
C12	0.026 (4)	0.029 (5)	0.065 (8)	0.003 (4)	0.019 (5)	0.013 (5)
C13	0.030 (4)	0.011 (3)	0.064 (7)	−0.001 (3)	0.021 (5)	0.008 (4)
C14	0.039 (6)	0.036 (5)	0.073 (9)	0.002 (4)	0.026 (6)	0.030 (6)
C15	0.098 (12)	0.043 (7)	0.092 (12)	0.033 (8)	0.075 (11)	0.042 (8)
C16	0.063 (9)	0.047 (7)	0.061 (9)	0.028 (7)	0.001 (7)	0.002 (7)
C17	0.034 (5)	0.025 (4)	0.052 (7)	0.006 (4)	0.017 (5)	0.018 (4)
C18	0.025 (4)	0.011 (3)	0.043 (5)	−0.003 (3)	0.018 (4)	0.005 (3)

Geometric parameters (Å, º)

Te1—O13	1.765 (6)	O12—V2[i]	2.039 (7)
Te1—O11	1.775 (6)	O1W—H1W1	0.853 (10)
Te1—O12	1.918 (7)	O1W—H2W1	0.845 (5)
Te1—O4	1.941 (7)	O2W—H1W2	0.849 (10)
Te1—O10	2.054 (7)	O2W—H2W2	0.849 (7)
Te1—O10[i]	2.080 (7)	O3W—H1W3	0.850 (10)
Te1—V4	3.095 (2)	O3W—H2W3	0.844 (7)
Te1—V3	3.105 (2)	O4W—H1W4	0.850 (10)
Te1—V1	3.161 (2)	O4W—H2W4	0.849 (10)

Te1—V2	3.1740 (18)	N1—C7	1.348 (13)
Te1—Te1ⁱ	3.220 (3)	N1—C9	1.366 (11)
V1—O7	1.610 (6)	N1—H1	0.8600
V1—O3	1.764 (6)	N2—C10	1.283 (14)
V1—O2	1.840 (6)	N2—C18	1.372 (12)
V1—O12	2.010 (7)	N2—H2	0.8600
V1—O4ⁱ	2.037 (7)	C1—C8	1.373 (17)
V1—O10ⁱ	2.281 (7)	C1—C2	1.45 (2)
V1—V2ⁱ	3.101 (2)	C1—H1A	0.9300
V1—V3	3.107 (2)	C2—C3	1.328 (19)
V2—O5	1.603 (6)	C2—H2A	0.9300
V2—O14	1.793 (6)	C3—C4	1.408 (15)
V2—O6	1.849 (6)	C3—H3	0.9300
V2—O4	2.005 (7)	C4—C9	1.412 (13)
V2—O12ⁱ	2.039 (7)	C4—C5	1.423 (15)
V2—O10ⁱ	2.285 (7)	C5—C6	1.380 (17)
V2—V1ⁱ	3.101 (2)	C5—H5	0.9300
V3—O9	1.604 (7)	C6—C7	1.336 (16)
V3—O8	1.828 (6)	C6—H6	0.9300
V3—O3	1.864 (6)	C7—H7	0.9300
V3—O14	1.895 (6)	C8—C9	1.372 (14)
V3—O11	2.027 (7)	C8—H8	0.9300
V3—O10ⁱ	2.380 (7)	C10—C11	1.339 (16)
V3—V4ⁱ	3.117 (3)	C10—H10	0.9300
V4—O1	1.602 (7)	C11—C12	1.390 (19)
V4—O8ⁱ	1.822 (6)	C11—H11	0.9300
V4—O2ⁱ	1.851 (6)	C12—C13	1.379 (18)
V4—O6ⁱ	1.905 (6)	C12—H12	0.9300
V4—O13	2.013 (6)	C13—C14	1.413 (17)
V4—O10	2.387 (7)	C13—C18	1.420 (12)
V4—V3ⁱ	3.117 (3)	C14—C15	1.40 (2)
O2—V4ⁱ	1.851 (6)	C14—H14	0.9300
O4—V1ⁱ	2.037 (7)	C15—C16	1.34 (2)
O6—V4ⁱ	1.905 (6)	C15—H15	0.9300
O8—V4ⁱ	1.822 (6)	C16—C17	1.39 (2)
O10—Te1ⁱ	2.080 (7)	C16—H16	0.9300
O10—V1ⁱ	2.281 (7)	C17—C18	1.414 (16)
O10—V2ⁱ	2.285 (7)	C17—H17	0.9300
O10—V3ⁱ	2.380 (7)		
O13—Te1—O11	106.2 (3)	O11—V3—Te1	32.64 (17)
O13—Te1—O12	96.4 (3)	O10ⁱ—V3—Te1	42.03 (17)
O11—Te1—O12	96.9 (3)	O9—V3—V1	133.6 (3)
O13—Te1—O4	96.4 (3)	O8—V3—V1	81.44 (19)
O11—Te1—O4	96.4 (3)	O3—V3—V1	30.14 (18)
O12—Te1—O4	158.2 (3)	O14—V3—V1	122.84 (18)
O13—Te1—O10	88.1 (3)	O11—V3—V1	82.33 (18)
O11—Te1—O10	165.7 (3)	O10ⁱ—V3—V1	46.83 (17)

O12—Te1—O10	81.5 (3)	Te1—V3—V1	61.17 (5)
O4—Te1—O10	81.3 (3)	O9—V3—V4i	134.2 (3)
O13—Te1—O10i	165.8 (3)	O8—V3—V4i	31.3 (2)
O11—Te1—O10i	88.0 (3)	O3—V3—V4i	83.04 (19)
O12—Te1—O10i	81.8 (3)	O14—V3—V4i	83.61 (18)
O4—Te1—O10i	81.4 (3)	O11—V3—V4i	123.93 (18)
O10—Te1—O10i	77.7 (3)	O10i—V3—V4i	49.27 (17)
O13—Te1—V4	37.73 (19)	Te1—V3—V4i	91.29 (7)
O11—Te1—V4	143.9 (2)	V1—V3—V4i	61.01 (5)
O12—Te1—V4	90.4 (2)	O1—V4—O8i	104.6 (3)
O4—Te1—V4	89.0 (2)	O1—V4—O2i	104.4 (3)
O10—Te1—V4	50.4 (2)	O8i—V4—O2i	89.8 (3)
O10i—Te1—V4	128.1 (2)	O1—V4—O6i	103.4 (3)
O13—Te1—V3	144.19 (19)	O8i—V4—O6i	88.9 (3)
O11—Te1—V3	38.0 (2)	O2i—V4—O6i	151.5 (3)
O12—Te1—V3	89.5 (2)	O1—V4—O13	100.9 (3)
O4—Te1—V3	90.3 (2)	O8i—V4—O13	154.4 (3)
O10—Te1—V3	127.7 (2)	O2i—V4—O13	85.6 (3)
O10i—Te1—V3	50.0 (2)	O6i—V4—O13	83.4 (3)
V4—Te1—V3	178.06 (5)	O1—V4—O10	174.8 (4)
O13—Te1—V1	133.8 (2)	O8i—V4—O10	80.5 (3)
O11—Te1—V1	84.6 (2)	O2i—V4—O10	76.5 (2)
O12—Te1—V1	37.4 (2)	O6i—V4—O10	75.3 (2)
O4—Te1—V1	127.5 (2)	O13—V4—O10	74.0 (2)
O10—Te1—V1	85.7 (2)	O1—V4—Te1	133.3 (3)
O10i—Te1—V1	46.1 (2)	O8i—V4—Te1	122.0 (2)
V4—Te1—V1	119.73 (6)	O2i—V4—Te1	78.91 (19)
V3—Te1—V1	59.45 (5)	O6i—V4—Te1	77.73 (19)
O13—Te1—V2	133.6 (2)	O13—V4—Te1	32.45 (17)
O11—Te1—V2	84.3 (2)	O10—V4—Te1	41.54 (17)
O12—Te1—V2	127.7 (2)	O1—V4—V3i	136.0 (3)
O4—Te1—V2	37.1 (2)	O8i—V4—V3i	31.41 (18)
O10—Te1—V2	85.5 (2)	O2i—V4—V3i	81.97 (19)
O10i—Te1—V2	45.95 (19)	O6i—V4—V3i	82.23 (18)
V4—Te1—V2	118.35 (5)	O13—V4—V3i	123.07 (18)
V3—Te1—V2	60.30 (5)	O10—V4—V3i	49.08 (18)
V1—Te1—V2	91.45 (7)	Te1—V4—V3i	90.62 (7)
O13—Te1—Te1i	127.3 (2)	V1—O2—V4i	117.8 (3)
O11—Te1—Te1i	126.5 (2)	V1—O3—V3	117.8 (3)
O12—Te1—Te1i	79.3 (2)	Te1—O4—V2	107.1 (3)
O4—Te1—Te1i	78.9 (2)	Te1—O4—V1i	107.2 (3)
O10—Te1—Te1i	39.14 (19)	V2—O4—V1i	100.2 (3)
O10i—Te1—Te1i	38.5 (2)	V2—O6—V4i	117.7 (3)
V4—Te1—Te1i	89.55 (6)	V4i—O8—V3	117.3 (3)
V3—Te1—Te1i	88.53 (6)	Te1—O10—Te1i	102.3 (3)
V1—Te1—Te1i	60.25 (6)	Te1—O10—V1i	95.2 (3)
V2—Te1—Te1i	60.03 (5)	Te1i—O10—V1i	92.8 (3)
O7—V1—O3	105.3 (3)	Te1—O10—V2i	94.9 (3)

O7—V1—O2	102.9 (3)	Te1ⁱ—O10—V2ⁱ	93.2 (3)
O3—V1—O2	93.8 (3)	V1ⁱ—O10—V2ⁱ	167.0 (4)
O7—V1—O12	101.4 (3)	Te1—O10—V3ⁱ	169.7 (4)
O3—V1—O12	91.1 (3)	Te1ⁱ—O10—V3ⁱ	88.0 (2)
O2—V1—O12	152.9 (3)	V1ⁱ—O10—V3ⁱ	83.6 (2)
O7—V1—O4ⁱ	99.3 (3)	V2ⁱ—O10—V3ⁱ	85.1 (2)
O3—V1—O4ⁱ	154.0 (3)	Te1—O10—V4	88.0 (2)
O2—V1—O4ⁱ	88.6 (3)	Te1ⁱ—O10—V4	169.6 (4)
O12—V1—O4ⁱ	75.9 (3)	V1ⁱ—O10—V4	85.2 (2)
O7—V1—O10ⁱ	172.9 (3)	V2ⁱ—O10—V4	86.9 (2)
O3—V1—O10ⁱ	81.1 (3)	V3ⁱ—O10—V4	81.7 (2)
O2—V1—O10ⁱ	79.5 (2)	Te1—O11—V3	109.4 (3)
O12—V1—O10ⁱ	75.0 (3)	Te1—O12—V1	107.1 (3)
O4ⁱ—V1—O10ⁱ	74.0 (3)	Te1—O12—V2ⁱ	107.8 (3)
O7—V1—V2ⁱ	89.7 (2)	V1—O12—V2ⁱ	99.9 (3)
O3—V1—V2ⁱ	131.4 (2)	Te1—O13—V4	109.8 (3)
O2—V1—V2ⁱ	128.1 (2)	V2—O14—V3	117.5 (3)
O12—V1—V2ⁱ	40.4 (2)	H1W1—O1W—H2W1	115 (3)
O4ⁱ—V1—V2ⁱ	39.5 (2)	H1W2—O2W—H2W2	115 (3)
O10ⁱ—V1—V2ⁱ	83.69 (19)	H1W3—O3W—H2W3	117 (3)
O7—V1—V3	137.1 (3)	H1W4—O4W—H2W4	115 (3)
O3—V1—V3	32.04 (18)	C7—N1—C9	121.5 (8)
O2—V1—V3	82.40 (18)	C7—N1—H1	119.2
O12—V1—V3	87.9 (2)	C9—N1—H1	119.2
O4ⁱ—V1—V3	123.5 (2)	C10—N2—C18	123.0 (8)
O10ⁱ—V1—V3	49.57 (18)	C10—N2—H2	118.5
V2ⁱ—V1—V3	120.78 (6)	C18—N2—H2	118.5
O7—V1—Te1	136.4 (2)	C8—C1—C2	118.3 (11)
O3—V1—Te1	77.3 (2)	C8—C1—H1A	120.9
O2—V1—Te1	120.50 (18)	C2—C1—H1A	120.9
O12—V1—Te1	35.4 (2)	C3—C2—C1	120.7 (11)
O4ⁱ—V1—Te1	79.20 (19)	C3—C2—H2A	119.6
O10ⁱ—V1—Te1	41.10 (17)	C1—C2—H2A	119.6
V2ⁱ—V1—Te1	61.43 (5)	C2—C3—C4	121.1 (11)
V3—V1—Te1	59.39 (5)	C2—C3—H3	119.4
O5—V2—O14	105.6 (3)	C4—C3—H3	119.4
O5—V2—O6	104.4 (3)	C3—C4—C9	118.3 (10)
O14—V2—O6	93.3 (3)	C3—C4—C5	123.2 (10)
O5—V2—O4	100.4 (3)	C9—C4—C5	118.5 (9)
O14—V2—O4	91.4 (3)	C6—C5—C4	118.8 (10)
O6—V2—O4	152.5 (3)	C6—C5—H5	120.6
O5—V2—O12ⁱ	100.0 (3)	C4—C5—H5	120.6
O14—V2—O12ⁱ	153.2 (3)	C7—C6—C5	120.7 (11)
O6—V2—O12ⁱ	88.1 (3)	C7—C6—H6	119.6
O4—V2—O12ⁱ	76.0 (3)	C5—C6—H6	119.6
O5—V2—O10ⁱ	172.8 (3)	C6—C7—N1	121.6 (10)
O14—V2—O10ⁱ	80.5 (2)	C6—C7—H7	119.2
O6—V2—O10ⁱ	78.9 (2)	N1—C7—H7	119.2

O4—V2—O10ⁱ	75.1 (3)	C9—C8—C1	120.9 (11)
O12ⁱ—V2—O10ⁱ	73.5 (3)	C9—C8—H8	119.6
O5—V2—V1ⁱ	89.4 (3)	C1—C8—H8	119.6
O14—V2—V1ⁱ	131.6 (2)	N1—C9—C8	120.7 (9)
O6—V2—V1ⁱ	127.8 (2)	N1—C9—C4	118.7 (8)
O4—V2—V1ⁱ	40.3 (2)	C8—C9—C4	120.6 (9)
O12ⁱ—V2—V1ⁱ	39.7 (2)	N2—C10—C11	123.5 (11)
O10ⁱ—V2—V1ⁱ	83.49 (19)	N2—C10—H10	118.2
O5—V2—Te1	135.7 (3)	C11—C10—H10	118.2
O14—V2—Te1	77.17 (19)	C10—C11—C12	118.4 (12)
O6—V2—Te1	119.75 (19)	C10—C11—H11	120.8
O4—V2—Te1	35.8 (2)	C12—C11—H11	120.8
O12ⁱ—V2—Te1	78.9 (2)	C13—C12—C11	118.9 (10)
O10ⁱ—V2—Te1	40.88 (18)	C13—C12—H12	120.6
V1ⁱ—V2—Te1	61.38 (5)	C11—C12—H12	120.6
O9—V3—O8	102.9 (3)	C12—C13—C14	122.8 (9)
O9—V3—O3	103.6 (3)	C12—C13—C18	120.4 (10)
O8—V3—O3	91.2 (3)	C14—C13—C18	116.7 (11)
O9—V3—O14	103.5 (3)	C15—C14—C13	120.2 (10)
O8—V3—O14	90.2 (2)	C15—C14—H14	119.9
O3—V3—O14	151.8 (3)	C13—C14—H14	119.9
O9—V3—O11	101.9 (3)	C16—C15—C14	120.6 (13)
O8—V3—O11	155.2 (3)	C16—C15—H15	119.7
O3—V3—O11	84.1 (3)	C14—C15—H15	119.7
O14—V3—O11	83.0 (3)	C15—C16—C17	123.3 (14)
O9—V3—O10ⁱ	176.5 (3)	C15—C16—H16	118.3
O8—V3—O10ⁱ	80.6 (3)	C17—C16—H16	118.3
O3—V3—O10ⁱ	76.5 (2)	C16—C17—C18	116.1 (10)
O14—V3—O10ⁱ	76.0 (2)	C16—C17—H17	122.0
O11—V3—O10ⁱ	74.7 (2)	C18—C17—H17	122.0
O9—V3—Te1	134.5 (3)	N2—C18—C17	121.4 (8)
O8—V3—Te1	122.6 (2)	N2—C18—C13	115.7 (10)
O3—V3—Te1	77.75 (19)	C17—C18—C13	122.9 (9)
O14—V3—Te1	77.87 (18)		
C8—C1—C2—C3	−0.8 (19)	C18—N2—C10—C11	1.8 (17)
C1—C2—C3—C4	1.2 (19)	N2—C10—C11—C12	−2.8 (18)
C2—C3—C4—C9	0.2 (17)	C10—C11—C12—C13	3.2 (18)
C2—C3—C4—C5	176.4 (12)	C11—C12—C13—C14	−178.1 (12)
C3—C4—C5—C6	178.5 (12)	C11—C12—C13—C18	−2.8 (17)
C9—C4—C5—C6	−5.3 (18)	C12—C13—C14—C15	178.9 (12)
C4—C5—C6—C7	3 (2)	C18—C13—C14—C15	3.4 (18)
C5—C6—C7—N1	1 (2)	C13—C14—C15—C16	−3 (2)
C9—N1—C7—C6	−3.1 (18)	C14—C15—C16—C17	4 (2)
C2—C1—C8—C9	−1.1 (19)	C15—C16—C17—C18	−5 (2)
C7—N1—C9—C8	−177.4 (11)	C10—N2—C18—C17	−179.1 (10)
C7—N1—C9—C4	0.7 (16)	C10—N2—C18—C13	−1.1 (14)
C1—C8—C9—N1	−179.5 (11)	C16—C17—C18—N2	−176.9 (10)

C1—C8—C9—C4	2.5 (17)	C16—C17—C18—C13	5.2 (16)
C3—C4—C9—N1	179.9 (10)	C12—C13—C18—N2	1.7 (14)
C5—C4—C9—N1	3.5 (15)	C14—C13—C18—N2	177.3 (10)
C3—C4—C9—C8	−2.0 (15)	C12—C13—C18—C17	179.7 (10)
C5—C4—C9—C8	−178.4 (11)	C14—C13—C18—C17	−4.7 (15)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H2*W*1···O2ii	0.85 (1)	1.82 (1)	2.663 (8)	178 (1)
O1*W*—H1*W*1···O7	0.85 (1)	1.92 (2)	2.762 (9)	170 (4)
O2*W*—H1*W*2···O8iii	0.85 (1)	2.03 (2)	2.842 (10)	159 (4)
O2*W*—H2*W*2···O4*W*iv	0.85 (1)	2.10 (1)	2.952 (14)	177 (1)
O3*W*—H2*W*3···O4*W*	0.84 (1)	1.91 (1)	2.754 (14)	177 (1)
O3*W*—H1*W*3···O6iii	0.85 (1)	1.83 (2)	2.665 (11)	166 (6)
O4*W*—H2*W*4···O1*W*v	0.85 (1)	2.41 (3)	2.826 (12)	111 (3)
O4*W*—H1*W*4···O2*W*	0.85 (1)	2.26 (5)	2.836 (17)	125 (5)
N1—H1···O1*W*	0.86	1.85	2.700 (11)	172
N2—H2···O14	0.86	1.88	2.740 (9)	175
C5—H5···O6iii	0.93	2.29	3.180 (13)	160
C6—H6···O13vi	0.93	2.48	3.178 (14)	132
C7—H7···O1vii	0.93	2.56	3.350 (13)	143
C7—H7···O2*W*viii	0.93	2.57	3.296 (14)	135
C10—H10···O9ix	0.93	2.51	3.275 (13)	140
C14—H14···O4x	0.93	2.58	3.403 (14)	148
C17—H17···O5	0.93	2.60	3.411 (13)	146

Symmetry codes: (ii) −*x*, −*y*, −*z*+1; (iii) *x*+1, *y*, *z*+1; (iv) −*x*+2, −*y*, −*z*+2; (v) *x*+1, *y*, *z*; (vi) −*x*+1, −*y*+1, −*z*+2; (vii) *x*, *y*−1, *z*; (viii) *x*−1, *y*, *z*; (ix) −*x*+1, −*y*+1, −*z*+1; (x) −*x*+1, −*y*+2, −*z*+1.